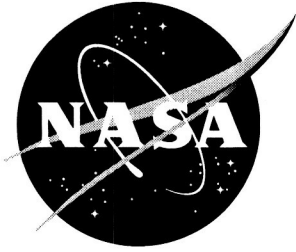


NASA/CR-2003-212400



Superplastic Forming/Adhesive Bonding of Aluminum (SPF/AB) Multi-Sheet Structures

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1.0 Background

Titanium superplastically-formed/diffusion-bonded (SPF/DB) panel structures are being installed on several aircraft, including the F-15E, C-17 and the T-38, and are being considered for commercial aircraft systems such as 737NG nose landing gear door. Part of the reason is reduced part count and cycle time. SPF/DB can result in sandwich structure costs that are 50% lower than conventional honeycomb construction. It also has the ability to produce tailored rib, and integral hard point and fastener through-hole, structures. Complex curvatures are more easily obtained. It is desirable to extend these advantages to aluminum-based systems.

Titanium alloys are amenable to the SPF/DB process because they can be readily diffusion-bonded at low contact pressures. Aluminum alloys do not exhibit this characteristic due to their stable Al_2O_3 surface film and low oxygen solubility. A viable method for selectively joining faying aluminum alloy surfaces during forming must be developed before multisheet SPF of aluminum panels can be produced. To this end, we have explored the use of an *in situ* adhesive film to provide the faying surface bond. This is termed "Superplastically-Formed, Adhesively-Bonded," (SPF/AB). It is important to emphasize that the adhesive is present during the forming process and not applied in a separate bonding step after forming. This permits bonding of regions that would normally be closed off to adhesive during post-forming bonding and eliminates secondary bonding steps after forming.

The current program followed on Task Order No. 38 of NAS1-20014 that ran May 1998 through June 1999. That program demonstrated the feasibility of superplastic forming/adhesive bonding (SPF/AB), but identified some areas that would require improvement to make it a success. The purpose of this task was to address these shortcomings, scale up the process to larger panels, and identify appropriate applications. This final report describes these efforts. The earlier program final report contains the technical background for the process and will not be repeated here. The basic process is illustrated in Figure 1.

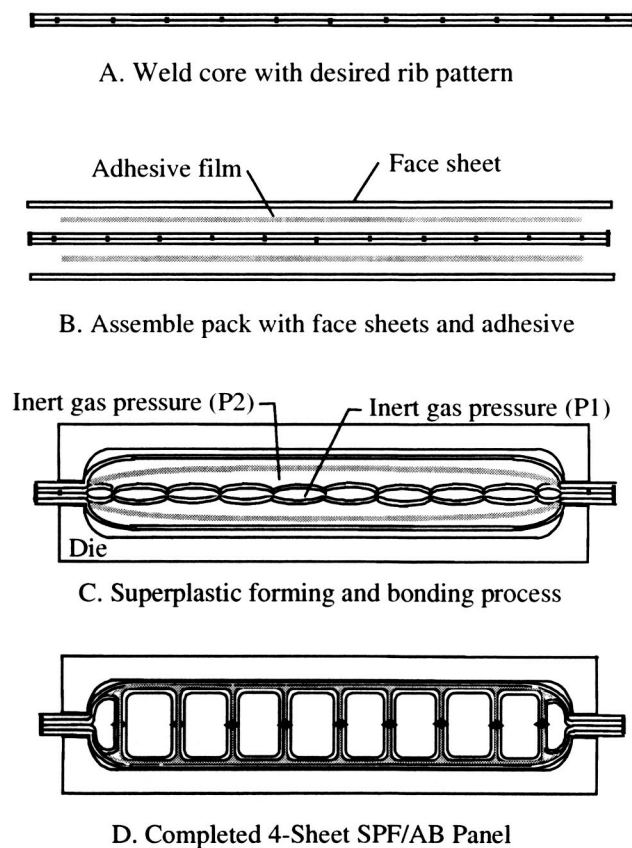


Figure 1. Basic SPF/AB Fabrication Approach (Adhesive Exaggerated)

2.0 Tooling, Equipment and Starting Materials

An existing 12" x 12" x 1.0" 321 S.S. die (50% split line) was utilized for all forming trials. Scale up was conducted with a 24" x 24" x 1.0" HN steel die (100% split line). Forming was conducted in a Murdock 225 T SPF press with a computer-controlled argon gas feed system. Laser welding was accomplished with a Convergent Energy 1700 W CO₂ continuous wave laser welder. Resistance welds were made using a Sciaky resistance seam welder. Friction stir welded SPF core packs were produced at Boeing's Huntington Beach welding laboratory using a 0.080" pin diameter tool.

Forty-three 39" x 78" x 0.050" sheets of unrecrystallized 8090 were procured from British Aluminum. One hundred 12" x 30" x 0.0017" sheets of unsupported LaRC 8515 polyimide adhesive film were procured from IMITEC Inc.

3.0 Laser Welding Conditions

Laser welding conditions were established on lap joints of the 8090 sheet in the etched and deoxidized condition. Good welds were obtained at 1440 W under helium cover gas at 40 cfh and 70" per minute travel speed. A 90 degree copper reflective focusing mirror with a 4" focal length was utilized.

4.0 Design of Experiments to Determine Optimum Process Conditions

A parametric DOE was established to identify the optimum set of processing conditions for forming and bonding 4-sheet SPF/AB panels. This was deemed necessary because, while the forming temperature range for 8090 is quite wide (600-975 °F), the maximum strain achievable depends on temperature. A moderate amount of strain (~75%) is required to fully form the fluted cells and close down on the adhesive on the internal panel surfaces. Since the LaRC 8515 adhesive limits the maximum time at temperature to about an hour, an optimal set of forming conditions needed to be identified that maximizes both bond strength and forming strain. The DOE was set up as a 1/2-fraction of a 4-variable, two-level factorial, as shown in Table I below. The principal independent variables were temperature, time, adhesive thickness and final pressure. Lap-Shear strength and rib forming radius were measured as dependent variables.

Table I. DOE Test Matrix for SPF/AB

| Run # | Variables | | | |
|-------|-----------|------------|--------------------------|----------------------|
| | Temp (F) | Time (min) | Adhesive Amount (sheets) | Final Pressure (psi) |
| 1 | 700 | 50 | 1 | 250 |
| 2 | 750 | 50 | 1 | 325 |
| 3 | 700 | 70 | 1 | 325 |
| 4 | 750 | 50 | 1 | 250 |
| 5 | 700 | 50 | 2 | 325 |
| 6 | 750 | 50 | 2 | 250 |
| 7 | 700 | 50 | 2 | 250 |
| 8 | 750 | 50 | 2 | 325 |

Two four-sheet forming trials, without adhesive, were conducted to help establish the appropriate range of conditions in the design-of-experiments (DOE) test matrix.

Core Pack Fabrication: Fifteen 2-sheet SPF core packs were laser-welded, utilizing the welding parameters previously described. A photograph of the core packs is presented in Figure 2. A limited number were used to develop the pressure cycle used for the DOE. The pressure cycle is the time-gas pressure path to reach the desired maximum pressure. These core-only packs were layered with adhesive and upper and lower face sheets to produce eight 4-sheet panels for the design-of-experiments (DOE) trials.

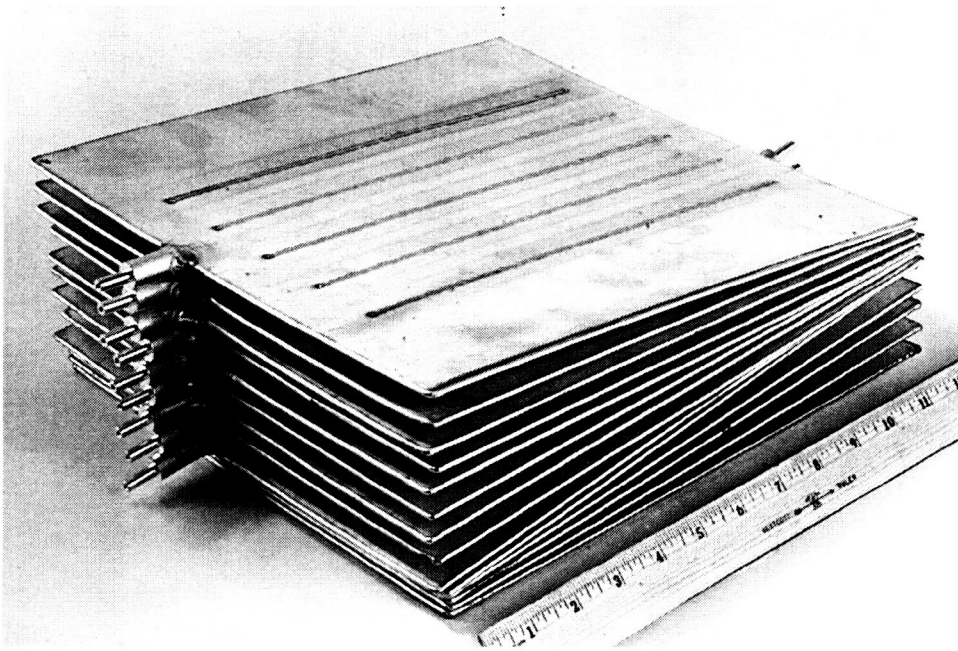
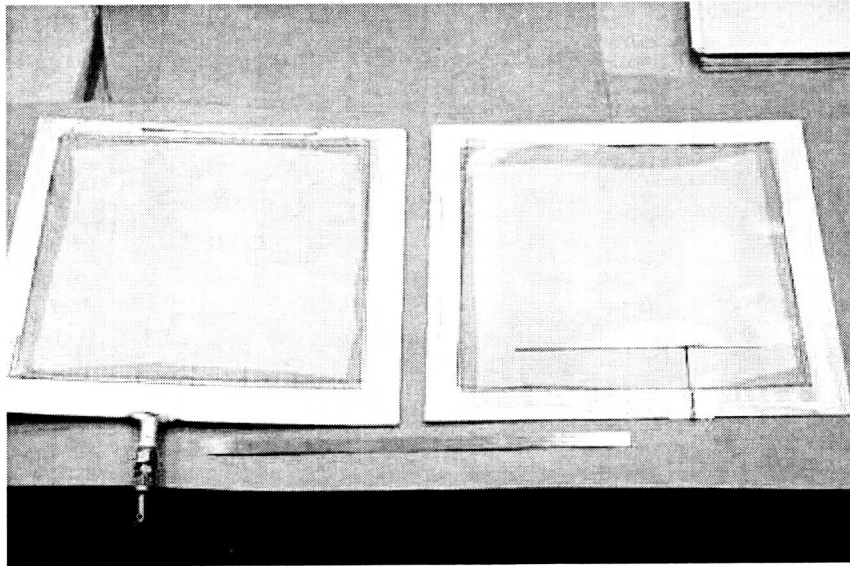


Figure 2. Laser-Welded 8090 Core Packs Ready for Superplastic Forming Trials

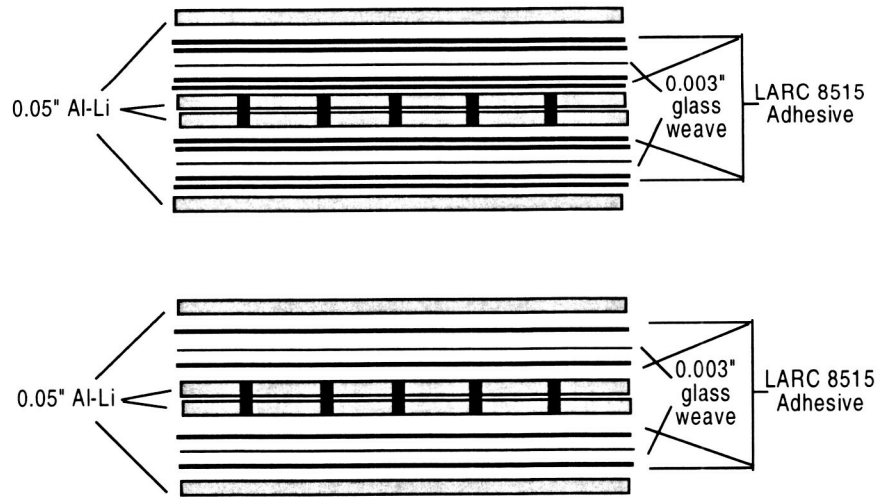
Surface Preparation: The Boegel-AM sol gel with BR X5 primer (Cytec) was identified as best for the LaRC 8515 adhesive system. Boegel-AM is an amino-functionalized silicon-zirconium sol gel. Cleaning and deoxidizing prior to primer application were accomplished with Brulin 815 GD and Amchem 6-16, respectively.

All welded cores and face sheet internal surfaces were prepared by this method for the DOE study.

Panel Fabrication: Eight panels were fabricated using the DOE parameters and the materials stackup shown in Figure 3a. Figure 3b shows the adhesive placement on the cores and Figure 4 shows the completed panels.



(a)



(b)

Figure 3. Layup of SPF/AB Panels (a) LaRC 8515 Adhesive on Al-Li 8090 Sheets; (b) Schematic of Panel Layers

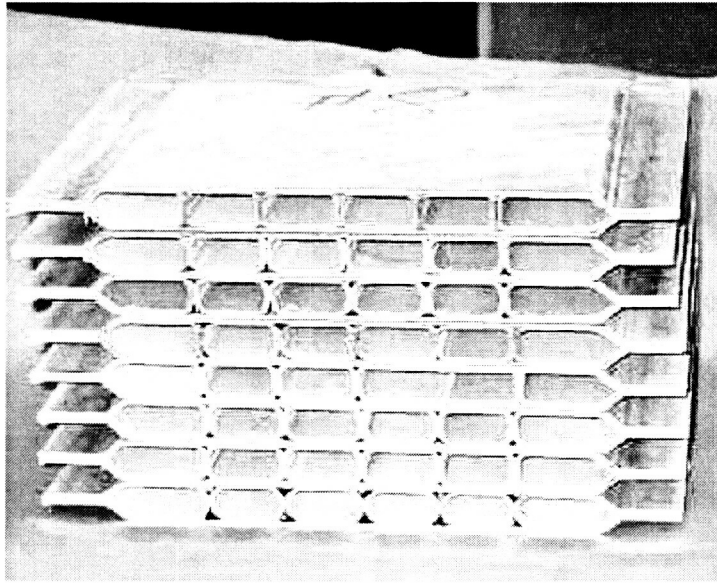


Figure 4. Stack of Eight 12" x 12" x 0.675" SPF/AB Panels

DOE Study Results: Single lap-shear specimens were excised from each panel (Figure 5) and tested to failure at room temperature. The effects of five factors (temperature, time, adhesive layers, pressure and side (top vs. bottom of panel)), and two-way interactions among the first four were considered in the DOE. The lap shear strengths of the bonds range from as low as 26.2 psi to as high as 767.2 psi, with an overall mean of 385.3 psi. The complete data set are given in the September 2000 Quarterly Report (NAS1-99070-MTPR-11). The main factors, in order of significance, were: 1) time-temperature interaction, 2) adhesive thickness and 3) time. After removing insignificant factors (at the 5% level), the following predictive model applies:

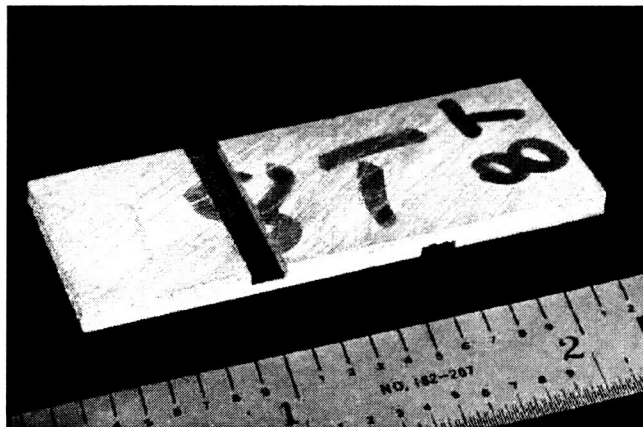


Figure 5. Single Lap-Shear Test Specimen for Panel #8 (Top).

The main factors, in order of significance, were: 1) time-temperature interaction, 2) adhesive thickness and 3) time. After removing insignificant factors (at the 5% level), the following predictive model applies:

$$\text{Bond Strength (lbs)} = 139.42 + 0.985(\text{Time} - 60) - 24.2(\text{adhesive} - 1.5) - 14.12 (\text{Time} - 60)(\text{Temp}-725)/250$$

As an example, for forming conditions at 750 °F, 70 minutes and one sheet of adhesive the predicted bond strength would be 147 psi. This model is statistically significant at 75% but explains only 15.5% of the observed variability. The residual variation would result in standard deviation of 42.23 lbs. Nonetheless, the model indicates that the bond strength is maximized for lower temperatures, shorter times and one sheet of adhesive.

The former are intuitive in that extended thermal exposures lead to degradation of the adhesive. The negative coefficient on the adhesive term indicates that excessive adhesive may be leading to lower bond strengths.

No significant variables were found to control the rib radius. However, it is clear from a knowledge of the forming process that higher temperatures and longer times should contribute to more extensive forming.

5.0 Bond Strength Improvements: The bond strengths measured on the DOE panels were about 50% of that of the coupons fabricated in the autoclave, and the DOE indicated that increasing time-temperature interactions were decreasing strength. Visual comparison of specimens from the DOE study indicated that the bonding process in the SPF chamber differed from that in the autoclave in that the adhesive in the bond line of most specimens appeared dark and oxidized. So, rather than simple thermal aging, it was postulated that the adhesive was suffering from air exposure.

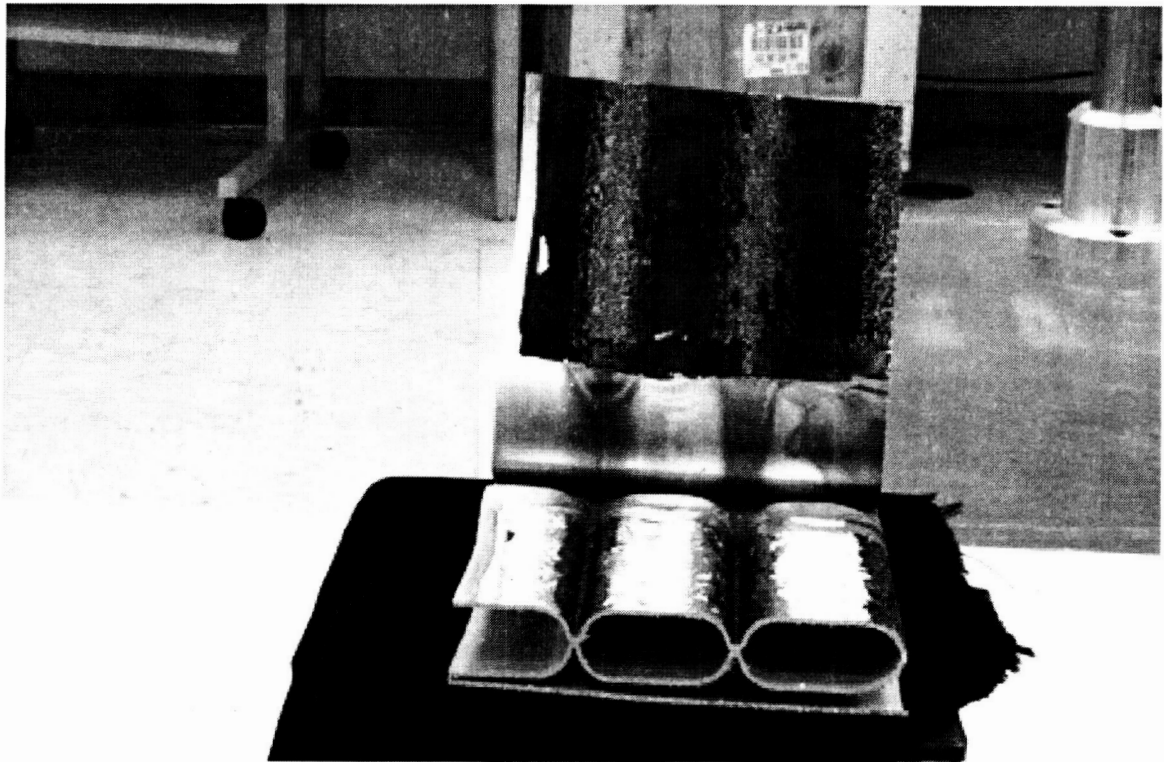


Figure 6. Oxidized Adhesive After Forming

This problem resulted in additional efforts and a revised statement of work. A revised SOW was generated to include subtasks to examine process changes in an attempt to reduce atmospheric exposure of the adhesive during forming. These included backfilling the adhesive zone with argon gas, and evacuating the adhesive zone.

Consequently, fourteen new 8090 core packs were fabricated by laser welding. A photograph of the laser welded cores is presented in Figure 7. After some preliminary

trials, three 4-sheet packs were fabricated under different conditions: (1) face sheet not sealed around perimeter (typical), with air allowed in area between core and face sheets (adhesive area); (2) face sheets sealed around perimeter and argon gas flowed through adhesive area during forming; and (3) face sheet sealed around perimeter and a vacuum held in adhesive area during the entire forming process.

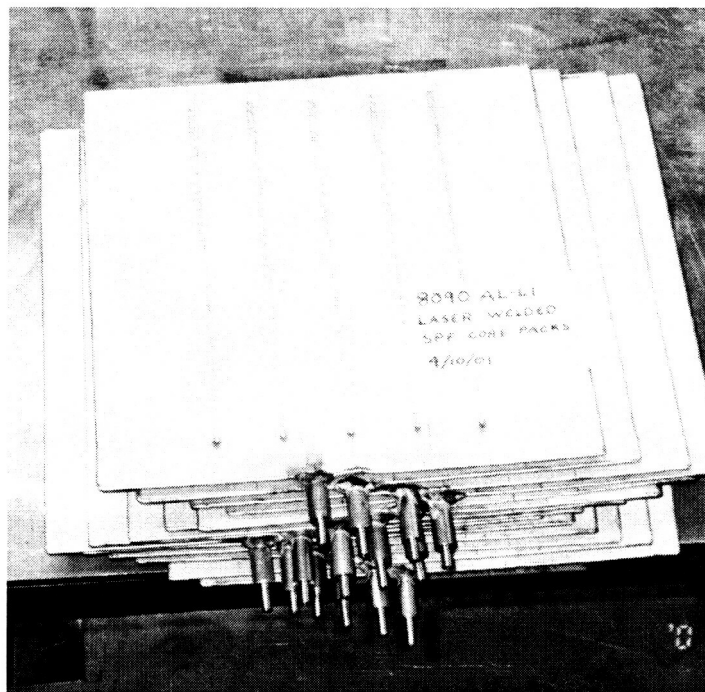


Figure 7. Fourteen laser welded cores, perimeter and gas inlet fitting TIG-welded.

Of the three environments, vacuum appeared to yield the best apparent bond, as determined by simple manual peel tests of the face sheets from the core.

Based on this experiment, five 12"x 12" panels were fabricated using the vacuum technique in order to evaluate bond strength. The packs were configured as in Figure 3. Although all five panels developed gas leaks prior to the completion of forming, adequate strain was attained in four to permit the extraction of four lap-shear test specimens per panel to characterize the bond strength. The results are shown in Table II.

Table II. Core Sheet to Face Sheet Tensile Lap-Shear Data

| Panel # | Test 1 (lbs) | Test 2 (lbs) | Test 3 (lbs) | Test 4 (lbs) | Mean (lbs) | Mean Shear Strength (psi) |
|---------|--------------|--------------|--------------|--------------|------------|---------------------------|
| 1 | -- | 468 | 1396 | 758 | 874 | 2331 |
| 2 | 1065 | 1140 | 1006 | 952 | 1041 | 2775 |
| 4 | 1094 | 1181 | 886 | 845 | 1002 | 2671 |
| 5 | 698 | 383 | 538 | 1005 | 656 | 1749 |

Specimen dimensions 2.5" long x 0.75" with 0.5" x 0.75" gauge sections.
Tests run at room temperature

These shear strengths are roughly four times that of specimens excised from DOE test panels, clearly indicating the benefit of the vacuum technique. Unfortunately, the panels were now frequently experiencing leaks in the weld regions prior to completion of the forming cycle. This prevented complete forming of the core flutes.

7.0 Friction Stir Welding (FSW) Fabrication of SPF Core Panels

Friction stir welding was investigated because it's ability to produce joints without melting, offering the potential to provide a sufficiently refined microstructure to produce superplastic joints. Five FSW fabricated cores were fabricated at Boeing's Huntington Beach welding laboratory. Figure 8 shows a completed 12"x 12" FSW core.

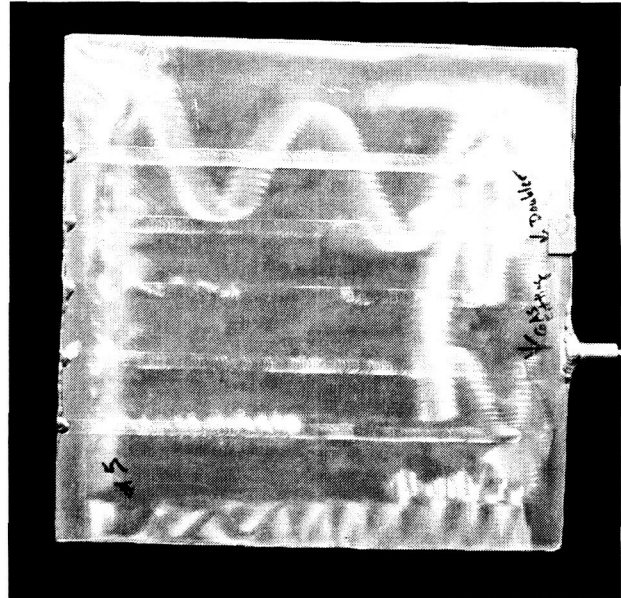


Figure 8. Friction Stir Welded Panel Core

Core-only trials were performed on all five FSW test panel cores at 750 degrees F with pressure cycles ranging from 1 to 3 psi/minute. All core-only test trials resulted in ruptures adjacent to the welds, in spite of extensive prior deformation of stirred material. It is postulated that the large joint width created by the FSW forced all deformation in the material immediately adjacent the joint (Figure 9), leading to early failure. FSW was abandoned after these exploratory trials.

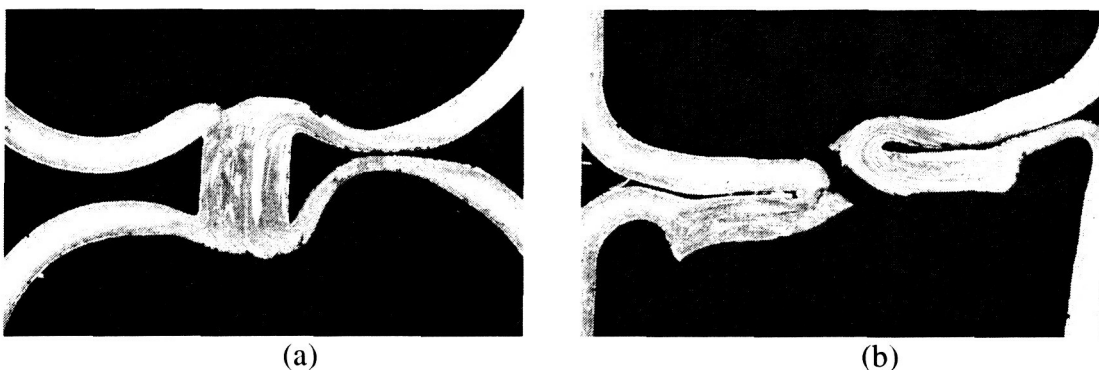


Figure 9: (a) Macrophotograph showing severe thin-out of ribs adjacent to FSW.
(b) Macrophotograph showing tearing of rib at FSW.

8.0 Panel Scaleup

Scaleup efforts involved fabrication of five 24" x 24" packs, using configurations similar to that described in sections 2.0 and 3.0. As in many of the forming trials, these panels also exhibited tearing at the laser weld joints prior to completion of forming. The panels are shown in Figure 10.

No additional panel fabrication trials were conducted in this task.

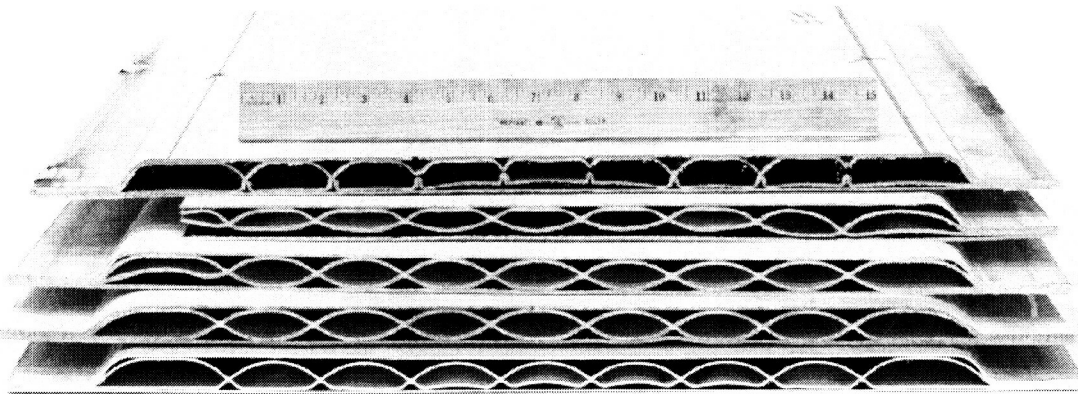


Figure 10. 24" x 24" Partially Formed SPF/AB Panels

9.0 Applications Development

One important subtask involved the identification of applications for SPF/AB technology, both to provide guidance for performance requirements, and to identify early-adopter customers.

Initially, commercial airplane applications were surveyed, such as control surfaces, access doors, underwing fuel access doors, and nose wheel doors. Two particular parts stand out for application to the SPF/AB process, one is a 747 underwing fuel access impact resistance door and the other is a 757 wing access door. The 747 door drawing is shown in Figure 11.

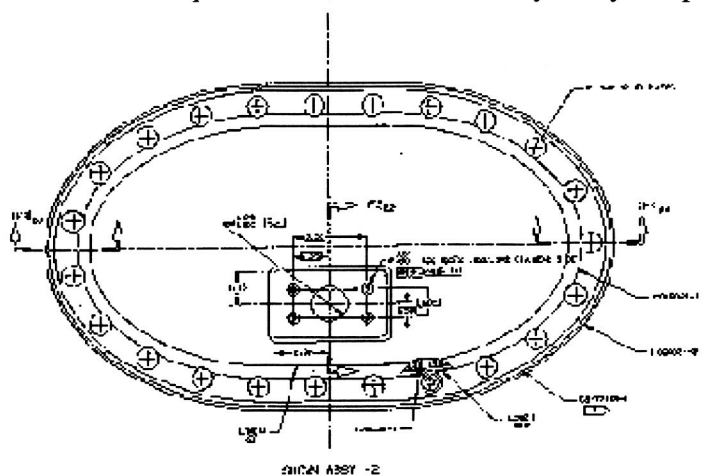


Figure 11. 747 Underwing Access Door (~22"x 14")

These were difficult to develop at such an early stage of development for this technology, largely because SPF/AB was competing with conventional metal-bond techniques that are inexpensive and well understood.

Lockheed Martin Corporation exhibited considerable interest in a possible satellite radiator concept after seeing Dr. Cotton's presentation of the SPF/AB work at the 1999 AeroMat conference. The schematic is shown in Figure 12. This resulted in extensive communication and a proposed radiator concept based on SPF/AB. However, it was deemed too early to commit to such an application.

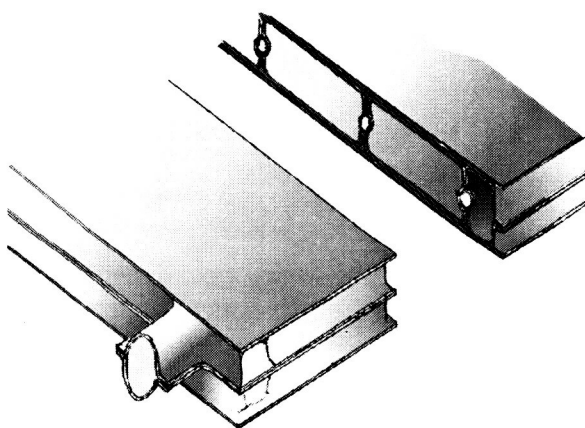


Figure 12. Lockheed Martin Space Radiator Concept

The most recent efforts to identify applications concerned space launch platforms, such as cryogenic tankage for the Delta IV. The program enlisted the assistance and engineers at Boeing-Huntington Beach. Unfortunately, the designers could not identify an application for SPF/AB sandwich structure on expendable or re-useable space type vehicles. Bond strength, panel life expectancy, and performance in cryogenic or elevated temperature environments were all cited as being concerns with adhesive bonding as the primary means of joining and fabrication.

10.0 Summary & Recommendations

Superplastic forming/adhesive bonding of aluminum panel structures was conceived as a process analogous to multisheet SPF/DB of titanium panel structures that would have advantages of reduced part count, reduced weight and lower cycle times. Two main technical challenges needed to be overcome: low adhesive bond strength and core weld failure during forming. The nontechnical challenges were the high cost of the constituent materials, and the non-unrelated task of identifying viable applications for the technology.

Notable progress was made in the arena of bond strength, which was improved by two orders of magnitude compared with early trials, and a factor of four in the past year. The bond strengths achieved in the SPF press approached those of commercial systems.

The problem of core weld failures was not solved in this effort. It is related to the change in microstructure due to welding which creates a microstructure that cannot be superplastic, yet has flow and fracture stresses in the same range as the flow stress for the unrecrystallized structure. Friction stir welding with smaller tool pins may hold promise in this area, and it is a recommendation of this team that this be pursued if this technology is further developed.

The principal challenge in identifying applications for SPF/AB was the competition from existing adhesively-bonded metal processes, such as honeycomb core panels. The perceived advantages of placing adhesive into formed faying bond surfaces and the elimination of secondary bond/cure steps were not deemed sufficient to warrant the applications investigated, and there is little reason to expect the price of SPF/AB to be less than current metal bond processes. It is recommended that, if SPF/AB development is continued, a lower cost alloy system be utilized.

11.0 Subtask & Deliverables Status

Because of the uncertainties of research and development, some of the subtasks defined in the revised statement of work could not be completed. For the benefit of the reviewer, these are tabulated below along with the current status.

| Subtask | Requirement | Status |
|---|---|---|
| 1. Procure Materials | Obtain 8090 and adhesive | Complete |
| 2. Develop Performance Requirements | Identify potential applications | Complete |
| 3. Subscale Process Development | a. Dev. surface prep. b. Core-only trials c. Make subscale panels (5 sections to NASA) d. Alternative skin zone atmosphere e. Increased adhesive f. Sol gel / PAA surface g. Resistance welding | a. Complete b. Complete c. Complete (1 panel to NASA) d. Complete e. Incomplete f. Incomplete g. Complete (substituted FSW) |
| 4. Process Scaleup | Six panels 2' x 2' w/ two delivered to NASA | Incomplete |
| 5. Subelement Mechanical Testing | Mechanical testing of scale up panels | Incomplete |
| 6. Application Development | Investigate applications / estimate costs if possible | Complete |
| 7. Program Management, Reporting and Travel | Reports, trip to NASA-LaRC for oral report | Complete |

The deliverables and schedule for the program are listed below.

1. Sections of develop panels from task 3 will be delivered to NASA (complete)
2. Quarterly report detailing lap shear data and edgewise compression data (complete, except edgewise compression data could not be collected)
3. Excess aluminum alloy sheet material (to be shipped)
4. Final report (complete)

| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
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| Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503. | | | | |
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| 13. ABSTRACT (Maximum 200 words) A significant fraction of airframe structure consists of stiffened panels that are costly and difficult to fabricate. This program explored a potentially lower-cost processing route for producing such panels. The alternative process sought to apply concurrent superplastic forming and adhesive bonding of aluminum alloy sheets. Processing conditions were chosen to balance adequate superplasticity of the alloy with thermal stability of the adhesive. As a first objective, an air-quenchable, superplastic aluminum-lithium alloy and a low-volatile content, low-viscosity adhesive with compatible forming/curing cycles were identified. A four-sheet forming pack was assembled which consisted of a welded two-sheet core separated from the face sheets by a layer of adhesive. Despite some preliminary success, of over 30 forming trials none was completely successful. The main problem was inadequate superplasticity in the heat-affected zones of the rib welds, which generally fractured prior to completion of the forming cycle. The welds are a necessary component in producing internal ribs by the "four-sheet" process. Other challenges, such as surface preparation and adhesive bonding, were adequately solved. But without the larger issue of tearing at the weld locations, complex panel fabrication by SPF/AB does not appear viable. | | | | |
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